

REMARKS

The claims have been amended to more particularly claim that which the Applicants believe is the invention and to correct inadvertent typographical errors. There has been no introduction of new matter.

Novelty and Inventive Step – WO/96/34829

In paragraph V.2. of the IPER, the Examiner alleged that Claim 1 lacked novelty under PCT Article 33(2) as being anticipated by WO/96/34829. The Examiner further states that that Claim lacks an inventive step under PCT Article 33(3) as being obvious over WO/96/34829.

The Prior-Art

The Examiner is referred to the text in the present specification on page 3, which reads as follows:

“In most methods for the synthesis of nanosize powders, two issues are particularly important; (1) the formation of fine, uniform size particles, and (2) the prevention of agglomeration. Nanoparticles of a uniform size can in principle be formed by carefully controlling nucleation and growth. Often, a variety of encapsulating methods are necessary to control the size of nanoparticles.”

“Agglomeration is often the result of Van der Waal's forces. The adverse effect of agglomeration on the sintering behavior of ceramic powders is well documented. Even in catalysis, the need for dispersed powders is well known. Often, supercritical drying can be used to obtain nonagglomerated powders. In liquid media, agglomeration can be suppressed through steric hindrance or through the manipulation of electrostatic interactions. The latter in polar liquids can be achieved by changing the pH and the ionic strength of the solution. Many techniques involve the use of surfactants. Often the powders which are nonagglomerated and well dispersed in a liquid, tend to agglomerate during the drying stage. Fortunately, methods such as slip-casting, gel-casting, pressure slip casting can be used to

achieve powder compaction in a wet state. Such has been demonstrated using submicron ceramic powders.”

“With the exception of milling, all the above methods are based on molecular synthesis of nanoparticles wherein the particles are built-up by atom-by-atom, or molecule-by-molecule, addition. Even in processes based on the decomposition of metal carbonyls, the growth of particles occurs by a layer-by-layer addition of atoms. As a result, a control of nucleation and growth is necessary to ensure the formation of nanosize particles. This often requires a very precise and difficult control of the reaction system, which renders the manufacture of the nanosize powder in large quantities impractical or impossible. In addition, the molecular synthesis processes are costly because of the relatively large capital expenditures required for the equipment to control the formation of only a small quantity of nanosize product.”

Based upon the English text of the abstract, WO '829 teaches the production of a suspension of particles formed in solvent in which the particles are barely soluble. The suspension is then processed to cause the particles to be compacted and/or to crystallize. This application teaches the control of agglomeration of powders, and is basically illustrative of the prior-art methods discussed above. The method of WO'829 is basically a system for controlling agglomeration by modifying the surface by a surface-blocking treatment. Stated in other words, the invention of WO'829 appears to be a method to control formation of insoluble agglomerations of the powder particles by treating the powder with a surface blocking substance in a solvent in which the powder is nearly insoluble.

While the Applicants do not have an English translation of the specification, the paragraph starting on page 1, line 26, appears to set forth the problem being solved by the WO '829 invention, which is that agglomerates forming the nanosize powder should be reducible to their primary size and that in many powder manufacturing processes agglomerations are formed that cannot be so reduced. (page 1, lines 31 to 34). Accordingly, the WO'829 invention is not directed to the specific process for the formation of the powders,

but the treatment of powders after being "... formed in the usually way." In the text starting on page 2, methods from the literature are reviewed, with a recitation that agglomerations are formed. For example, for hydrothermal synthesis, it is stated (here in translation) at page 2, lines 30-32, that, "Because the formation of agglomerates is generally not reversible, the potential of this technique at this time is used only with limitations." Likewise for other processes, the problem of the formation of agglomerates that are not easily reduced to the primary sized particles is discussed. The solution in the WO'829 invention is to suspend the particles in a solvent in which the powder is hardly able to dissolve and that contains a surface-blocking agent. Surface-blocking (oberflächenblockierende) substances are disclosed on page 8, starting at line 20, and include substances the bond with the surface. These surface blockers (Oberflächenblocker) include various organic compounds, including certain "poly" acids (säuren), diamines, aminoacids, long chain alcohols, cellulose derivatives, etc. Specific examples listed in the paragraph starting on line 35, page 8, include various commercially available fatty acids, polyethylene-oxide derivatives, and fatty acid-polyglycol esters.

Present Invention.

In contrast to the WO '829 process, the present invention does not apply to known methods for making a powder, but the present invention is a new and unobvious method for the initial production of the powder from raw materials. Referring to the specification at page 4, starting at line 15,

"In order to overcome the problems associated with molecular synthesis and milling to form nanosize powders, the present invention presents an alternative approach for the synthesis of nanosize powders. In the present invention, a precursor inorganic compound is formed from which the unwanted component is leached away so that a fine, nanosize powder is left as a residue. Thus, the present invention is based on molecular decomposition, rather than molecular synthesis, or deposition."

In the method of the invention, undesired reactions occurring after formation of the powder particles that grow or agglomerate the particles are essentially nonexistent. In the present invention the solid-phase material (the non-soluble constituent) forming the nanosize particles exists already in the precursor. The non-soluble and fugitive constituents are formed together as the precursor, and a solvent removes the soluble solid phase of the fugitive constituent, leaving the non-soluble constituent behind. Because the precursor and the non-soluble constituent are not very soluble in the solvent, they are not dissolved, and thus, there is little possibility that these materials will recrystallize from solution to grow or agglomerate small particles. In addition, the fugitive constituent is so soluble, that little or none of the dissolved fugitive constituent precipitates and recrystallizes.

In a very rough sense, a metaphor for the present process is a lost wax metal casting process on a molecular scale, where a fugitive (wax) material is removed to leave a solid non-removable, non-soluble material. As indicated in the paragraph starting on page 6, line 8, the fugitive and non-soluble constituents in the precursor exist together as a compound or alloy on the molecular level. Accordingly, when the fugitive material is removed, voids left by the removal of the fugitive material (which are of a molecular scale) leave the remaining solid residue in a form that is easily reduced to nanosize particles. Since these particles are then in an environment that is basically non-reactive to the non-soluble substance of the particles, there is little tendency for the particles to grow or to agglomerate together. This contrasts with certain prior-art processes in which the particle are formed in a growth environment and remain in an environment that is to some extent chemically active toward surface of the particles, causing the particles to agglomerate or grow larger.

Summary

In contrast to the present invention, the solution of the agglomeration problem presented by WO '829 is not to change the basic particle growth processes of the prior-art, but to decrease the reactivity of the particle surface toward the chemically active environment by introducing an organic surface-blocking agent. The present invention differs completely from this approach. The method of the invention has solved the problem of agglomeration and continued particle growth by not growing the particles. Instead, the particles are manufactured as a residue of a decomposition. Thus, tendency to grow or agglomerate is suppressed.

It is believed that the present invention is both novel and has inventive step (is not obvious) over the WO/96/34829 reference. For this reason, the Examiner is respectfully requested to find the present claims patentable over this reference and allow the claims.

Novelty and Inventive Step – Chow et al.

In paragraph V.2. of the IPER, the Examiner alleged that Claim 1 lacked novelty under PCT Article 33(2) as being anticipated by Chow et al. The Examiner further states that that Claims 21 and 22 lack an inventive step under PCT Article 33(3) as being obvious over Chow et al.

In Paragraph Chow et al. discloses a method where a metal precursor is mixed and with and dissolved in an alcoholic solvent. The mixture is heated, reducing the metal precursor in solution to form particles as a metal precipitate. In this method, the precipitate particles are clearly grown as crystallites from a solution. Accordingly, the size of the crystallites formed is controlled by process parameters, such as, for example, concentration, (col. 3, line 12), reaction time (col. 3, lines 29), and ph (col. 3, line 65). For the purposes of the present discussion, this reference is illustrative of the prior-art particle growth processes, where particles are grown from a solution and attempt is made to control the size by

controlling the reaction conditions and length. This reference does not disclose and does not suggest the present invention for many of the reasons already discussed in the above section.

The disclosure of Chow is for the molecular synthesis of particles, and does not resemble the present invention where particles are a residue formed by decomposing a precursor of a fugitive and non-soluble constituent. There is no disclosure or suggestion in Chow to remove a fugitive constituent from a precursor and leave a non-soluble residue of small particles.

It is believed that the present invention is both novel and is not obvious over the Chow et al. reference. For this reason, the Examiner is respectfully requested to find the present claims patentable over this reference.

Summary

The Examiner is referred to the discussion starting on page 22, under the heading "Advantages of the Method of the Present Invention..." Therein are discussed some advantages over the prior-art. (1) The precursor materials of the present invention are homogeneous solids, such as solutions or compounds. Accordingly, when the fugitive material is removed the constituents of the remaining non-dissolved residue are intimately mixed. (2) Because there is a regular and uniform distribution of the fugitive constituent in the precursor material, when it is removed the remaining non-dissolved material should have a uniform particle size. (3) There is negligible growth of the nanosize particles because the non-soluble residue is essentially non-soluble in the solvent. It is believed that these advantages are not suggested over the cited references, and the present invention is accordingly patentable.

The applicant believes the present claims to be allowable under sections 35 U.S.C. §102 and, 35 U.S.C. §103. In addition, it is believed that the amended claims meet the

requirement of 35 U.S.C. §112. Accordingly, the Examiner is respectfully requested to allow all the present claims, as amended.

Respectfully submitted;

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VERSION WITH MARKINGS TO SHOW CHANGES MADE

Claims

1. (Amended) A process for forming a nanosize ceramic powder comprising:
forming a precursor ceramic material comprising a fugitive constituent and a non-soluble constituent in a single phase;

contacting the precursor material with a selective solvent to form a solution of the fugitive constituent in the solvent and a non-dissolved residue of the non-soluble constituent,

[the precursor material sufficiently reactive with the solvent to form the solution of the fugitive constituent in the solvent and form the nondissolved residue of the non-soluble constituent,

← Same as 1

^{NAB}
the precursor material and the non-soluble residue sufficiently insoluble in the solvent such that there is essentially no [insufficient] precursor material and non-soluble residue in the solution that will [to] deposit and precipitate upon the residue of the non-soluble-constituent,

the fugitive constituent being sufficiently soluble in the solvent such that [the precursor reacts with the solvent to form a] from the solution of the fugitive constituent ^{1/2} essentially no fugitive constituent will deposit and precipitate [without precipitation and deposition of fugitive constituent] upon the residue of the non-soluble constituent [in the form of nanosize particles],

removing the [selective solvent] solution of the fugitive constituent from the residue to form a nanosize powder of the residue of the non-soluble constituent.

21. (Amended) A process for forming a nanosize metallic powder comprising:
forming a precursor metallic material comprising a fugitive metal constituent and a
non-soluble metal constituent in a single phase;

contacting the precursor material with a selective solvent to form a solution of the
fugitive constituent and a non-dissolved residue of the non-soluble constituent,

the precursor material sufficiently reactive with the solvent to form the solution of the
fugitive constituent in the solvent and form the non-dissolved residue of the non-soluble
constituent,

same as

^{NAB}
the precursor material and the non-soluble residue sufficiently insoluble in the solvent such
that there is essentially no [insufficient] precursor material and non-soluble residue in the
solution that will [to] deposit and precipitate upon the residue of the non-soluble-constituent,

the fugitive constituent being sufficiently soluble in the solvent such that [the precursor reacts
with the solvent to form a] ^{or forward} from the solution of the fugitive constituent ¹¹² essentially no fugitive
constituent will deposit and precipitate [without precipitation and deposition of fugitive
constituent] upon the residue of the non-soluble constituent [in the form of nanosize
particles],

removing the [selective solvent] solution of the fugitive constituent from the residue
to form a nanosize powder of the residue of the non-soluble constituent.